

## SHOP NOTES

*These are "how to do it" papers. They should be written and illustrated so that the reader may easily follow whatever instruction or advice is being given.*

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### A simple fabrication method for nanometer-scale thin-metal stencils

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We describe a simple fabrication method for the production of thin-metal stencils using the overlayer film from lift-off patterning. While such stencils have been fabricated before using other techniques, this method has several advantages. The advantages include the fact that this method requires only the capability to perform standard lithography and thin-film deposition, but does not require any dry etching or other sophisticated capabilities. In addition, by using the negative of the lithographically defined pattern, we show that a small number of trenches of lateral size down to 100 nm, in an otherwise continuous stencil, can be produced using electron-beam lithography with very little beam rastering necessary (i.e., small exposure times). We also argue that any group with access to simple photolithography and thin-film deposition facilities should be able to make these stencils, with openings much smaller than the lithography resolution limit. [S0734-211X(97)00202-3]

Self-supported stencils (continuous sheets with a defined set of openings) are of potential technological importance for several areas. In the integrated circuit industry, these applications include masks for thin-film fabrication procedures (such as deposition, ion milling,<sup>1</sup> or ion implantation) that can be enhanced by the use of a stencil mask. Other applications of metal or nonmetal stencils include fabrication of devices for biomedical applications<sup>1,2</sup> and innovative biological materials.<sup>3</sup>

There are previous reports of fabrication of thin-metal stencils by various procedures. One group has used standard thin-film lithographic techniques to make gold gratings on top of SiN<sub>x</sub> membranes for x-ray astronomy.<sup>4</sup> One important criterion for these gratings is that they be phase coherent over a large number of periods. Driven in part by this criterion, the process to make these gratings is fairly lengthy; it involves a holographic photolithography sequence followed by shadow deposition of SiO<sub>2</sub>, oxygen reactive-ion etch, gold electroplating, and oxygen-plasma etching.<sup>4</sup> A later extension of this procedure<sup>5</sup> to produce free-standing gratings involved a subsequent superposition of two additional (larger) gratings for mechanical support, followed by etch removal of the underlying membrane.

Another procedure to make thin-metal stencils involves the production of replicas from arrays of nanochannel glasses.<sup>6</sup> The glass arrays are made by repeated stacking and drawing of glass fibers, followed by cleaving the stack to expose the cross section. Wafering, polishing, and acid etching produce a cross section with a controlled porous structure when the original array is formed from fibers with etchable and nonetchable components. To make the metal replica, a

thin-film bilayer (an easily dissolved buffer layer such as aluminum, followed by the metal of choice) is then deposited on the end surface of the array. Finally, the buffer layer is etched away in solution, and the replica can then be picked up for use.

These replicas have been made in thin films of tungsten, molybdenum, platinum, and gold, with packing densities as high as  $3 \times 10^9$  voids/cm<sup>2</sup>, and are obviously well-suited for the production of large uniform arrays. They require access to a draw tower, however, and they are not as well-suited for the production of custom lateral patterns due to the sophisticated procedure needed to modify the pattern. In addition, they may not be well-suited for the opposite limit of a small number of specifically placed holes or for continuous lines or large holes.

In this work, we report on a simple process to make thin-film stencils using electron-beam lithography (EBL), which consists of preparation of a bilayer resist, exposure to a rastered electron beam, and development; this is followed by deposition of aluminum. The stencil is then conveniently lifted off by soaking in acetone, and can then be recaptured on any desired substrate (a solid or one with open spaces). Although this process, which has succeeded in making trenches with sizes down to 100 nm, does require access to EBL, we believe that the analogous process using photolithography should allow any group to make similar stencils, although perhaps with a larger minimum feature size.

The fabrication process starts with the creation of a bilayer resist pattern (refer to the schematic in Fig. 1) consisting of about 0.5  $\mu\text{m}$  of co-poly-methylmethacrylate (PMMA) with about 0.1  $\mu\text{m}$  of PMMA on top. The reason for using this bilayer is that the co-PMMA has a much lower dose threshold than the PMMA, and thus provides the under-

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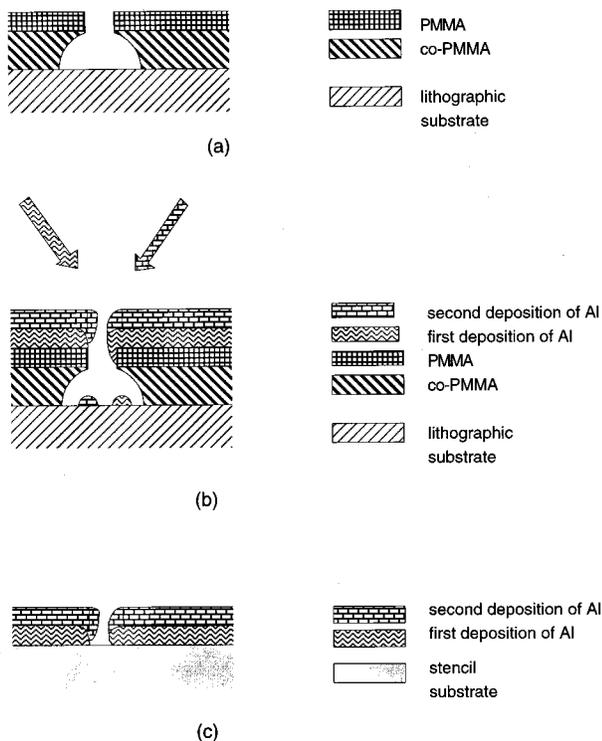


FIG. 1. Schematic of the procedure: (a) after lithographic exposure and development of the bilayer resist; (b) after deposition of metal at two different angles, yielding asymmetric film profiles; (c) after liftoff and recapture of the stencil onto a different (possibly with open holes) substrate. Note that the dual-angle deposition yields the potential for a stencil opening substantially smaller than the lithographically produced one.

cut shown in Fig. 1. We made this bilayer by standard resist spinning and baking. The EBL exposure used about  $100 \mu\text{C}/\text{cm}^2$  for areal doses and  $2 \text{ nC}/\text{cm}$  for linear doses. For each of the structures shown herein, the writing time was a few seconds. We developed the written pattern for 2 min in a solution of 1:3 MIBK:IPA (methyl isobutyl ketone:isopropyl alcohol). This procedure results in the structure indicated schematically in Fig. 1(a).

We then deposited Al by resistive heating of an Al shot in a vacuum of about  $10^{-5}$  Torr, with film thicknesses between 25 and 400 nm; in several cases the deposition was achieved from two separated sources and, thus, from different angles. In addition, we sometimes allowed a controlled oxidation of the first deposited layer by introducing about 100 mTorr of  $\text{O}_2$  for 5 min. This entire procedure (bilayer resist, dual-angle deposition with an intervening oxidation) was developed for and is extensively used for the production of ultrasmall Al/ $\text{AlO}_x$ /Al tunnel junctions.<sup>7</sup> We have indicated schematically the structure after deposition in Fig. 1(b) for the situation of a dual-angle deposition. Finally, we lifted off the stencil by soaking the substrate in acetone for 2 h. The films come off the substrate in sections with sizes between a few mm and 2 cm (2 cm was the largest substrate size used). A convenient way to cut the film is by scratching either the resist layer before deposition or the film after deposition; either way results in a clean break of the lifted-off film. We then “captured” the stencils onto glass slides, Si wafers, or

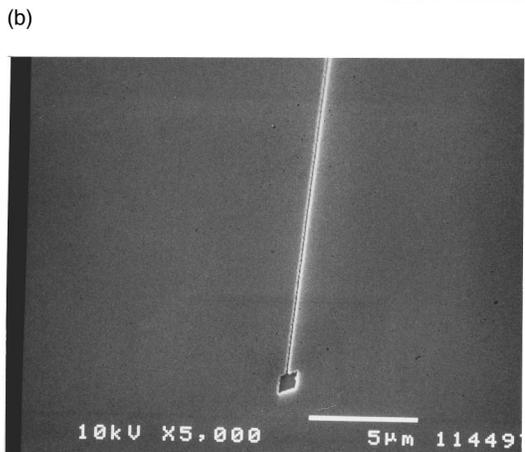
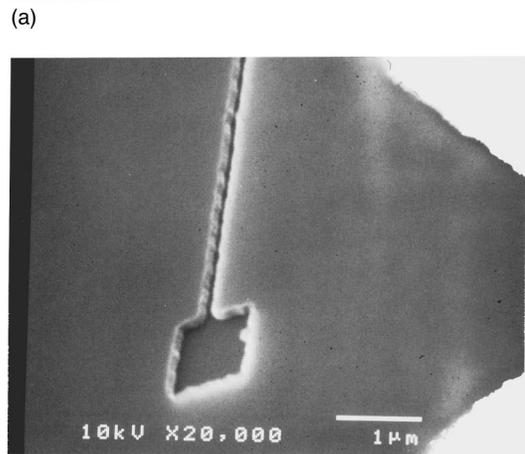
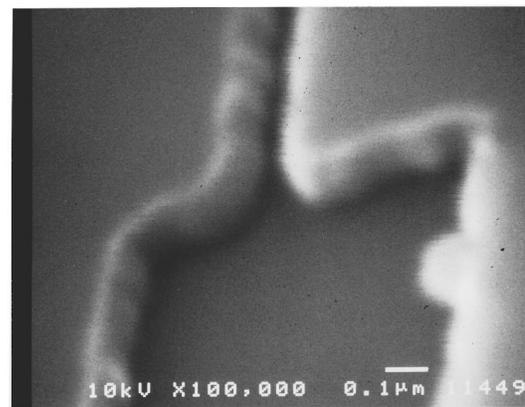


FIG. 2. SEM micrographs of a 100-nm-thick stencil captured onto a Si wafer, at an oblique tilt of about  $50^\circ$ , and three different magnifications. Note the well-formed nature of the square hole and trench; the firm edges, and that the stencil has flattened against the solid substrate. Note also the absence of any pinholes or other imperfections.

transmission electron microscopy (TEM) copper grids by pulling the film out of the solution at the same time as the wafer or grid.

Films with thicknesses of 25 or 50 nm were somewhat fragile, and sometimes lifted off in pieces as small as a few mm. Handling the pieces in acetone with a pair of tweezers would sometimes cause them to rip. Films between 100 and 400 nm thick were quite robust; we generally succeeded in

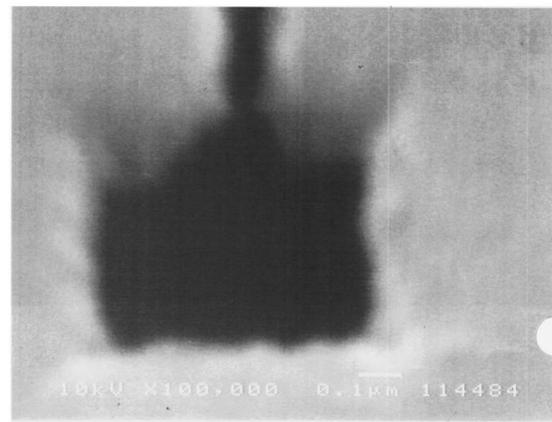
simply peeling them off the substrate with a pair of tweezers, and we handled them in the acetone without any damage. The upper limit of 400 nm was set only by the maximum convenient deposition thickness available.

An example of a stencil produced in this way is pictured in Fig. 2. This stencil was 100 nm thick, with an intervening oxide layer of about 2 nm thickness, and was captured from the acetone onto a Si substrate for convenient scanning electron microscope (SEM) examination. The area shown was produced by an EBL exposure of a square hole (edge length) 1  $\mu\text{m}$ , and a single pass line drawn from this hole. These micrographs were taken at an oblique tilt of about 50° so that the film edges might be examined.

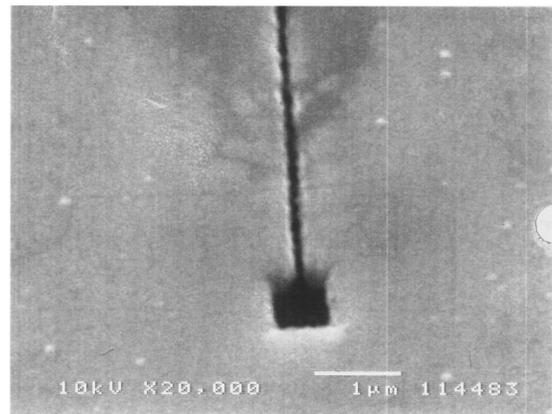
Several facts are obvious from these pictures: (1) except for the small knob in the right-hand edge of the hole, the holes in the stencil are the exact negative of the rastered EBL pattern, with well-formed, vertical edges. We note that the ripples along the hole and trench edges are an artifact of the 60 Hz wavering of the rastered electron beam, and are not intrinsic to the stencil production. (2) Over the area of approximately 20  $\mu\text{m}$  by 30  $\mu\text{m}$ , there are no observable pinholes or other imperfections. Examination of many stencils at lower magnification (not shown) demonstrated the absence of pinholes over areas of at least 0.1  $\text{mm}^2$ . (3) As indicated by the substrate visible through the square hole, the stencil has flattened against the substrate to within 10–20 nm (recall that the film thickness here is 100 nm).

Since most applications of such stencils involve passage of particles (ions, atoms, light) through the holes, it is important to show that these stencils can also be captured onto substrates that have open areas. Figure 3 shows SEM micrographs of a stencil captured onto a TEM grid (square copper mesh with wire widths of 6  $\mu\text{m}$ , and open squares with edge length of 20  $\mu\text{m}$ ). We recorded these pictures with the grid tilted at 50°; in this case the tilt was aligned with the trench. Examination of larger holes (not shown) indicates that the stencil lies fairly flat against the grid, with no tearing or breaking over the open areas. Again, the rastered pattern that produced these holes was a line between two square holes. In this case, the “line” was actually an area 0.3  $\mu\text{m}$  in width.

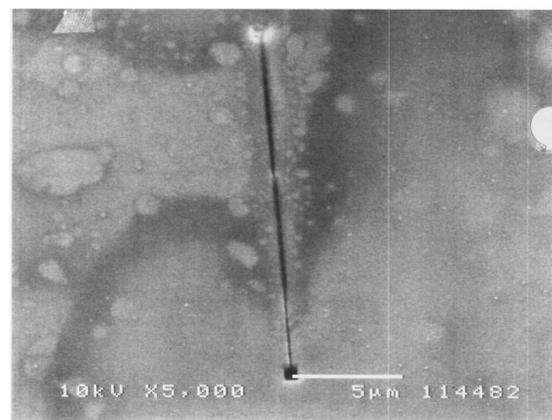
We can see several important facts from these micrographs. (1) As in the case on the solid substrate, the holes in this self-supported stencil are well-formed negatives of the written patterns. (2) The low-magnification picture [Fig. 3(c)] again shows no pinholes over an area of 20  $\mu\text{m}$  by 40  $\mu\text{m}$ . (3) The high-magnification picture shows a useful feature of the dual-angle deposition: as indicated schematically in Fig. 1(b), the holes in the bilayer resist can result in substantially smaller holes in the final stencil. The trench shown here was produced from a resist hole 0.3  $\mu\text{m}$  wide. We can see that the edge profile is beveled from about 0.27  $\mu\text{m}$  on the bottom side of the stencil to about 0.08  $\mu\text{m}$  on the top side. Note the resemblance between this profile and the schematic in Fig. 1(c). Thus, by appropriate deposition geometry, workers can make stencil holes or trenches with openings substantially smaller than the lithographic resolution. This fact forms the basis for the contention that workers with



(a)



(b)



(c)

Fig. 3. SEM micrographs of a 400-nm-thick stencil captured onto a TEM grid, at a tilt of 50° aligned along the trench. The areas shown are sitting over open areas in the grid, and the stencil is self-supported. The edge profile in the high-magnification picture (a) shows the potential for forming openings in the stencil that are much smaller than the original lithographic opening; in this case, the original trench was about 0.3  $\mu\text{m}$  in width. Again note the absence of pinholes or other imperfections.

access to photolithographic capabilities may still only be able to produce stencils with holes substantially smaller than the resolution limit. We note here that this will not be as effective as for EBL (i.e., in Fig. 3 we see about a factor of

3 decrease in the trench width) since the ratio of film thickness to lithography resolution will be smaller for photolithography. In addition, it appears that nonuniformities in the resist (the ripple along the trench arises from 60 Hz noise during the EBL exposure) may be amplified by the beveling (compare the ripples in Figs. 2 and 3).

In this note, we have demonstrated that a simple, convenient (typically requiring a few hours of worker time, and about one day total) process exists to make self-supporting thin-metal stencils that can be captured from acetone onto solid or perforated substrates. This procedure works surprisingly well due to the excellent liftoff achievable with the bilayer resist used. The stencil holes can be made substantially smaller than the original lithographic pattern, thus affording the potential for achieving nanometer-scale stencil holes with simple photolithographic facilities. The stencils produced are mechanically robust, with dimensions up to at least 1 or 2 cm, and are pinhole free over areas of at least 0.1

mm<sup>2</sup>. This process is particularly well-suited for applications that need small numbers of precisely located and precisely shaped holes.

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<sup>1</sup>R. Dizon *et al.*, *J. Microelectromech. Sys.* **2**, 151 (1993).

<sup>2</sup>S. Ufer *et al.*, *Proceedings of the 16th Annual International Conference of the IEEE Engineering in Medicine and Biology Society. Engineering Advances: New Opportunities for Biomedical Engineers* (IEEE, New York, 1994), p. 840.

<sup>3</sup>G. M. Chow *et al.*, *Mater. Sci. Eng. A* **158**, 1 (1992).

<sup>4</sup>M. Schattenburg *et al.*, *Opt. Eng.* **30**, 1590 (1991).

<sup>5</sup>E. E. Scime *et al.*, *Appl. Opt.* **34**, 654 (1995).

<sup>6</sup>D. H. Pearson and R. J. Tonucci, *Science* **270**, 68 (1995).

<sup>7</sup>T. A. Fulton and G. J. Dolan, *Phys. Rev. Lett.* **59**, 109 (1987).